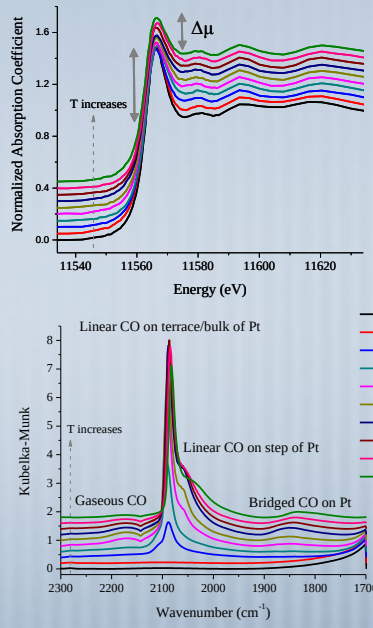
Combined In Situ X-ray Absorption and Diffuse Reflectance Infrared Spectroscopy: A Novel Tool for Catalytic Investigations

N. Marinkovic[1], Q. Wang[3], L. Barrio[2], S.N. Ehrlich[1], C. Cooper[3], A.I. Frenkel[3]
[1]University of Delaware; [2]Brookhaven National Laboratory; [3]Yeshiva University

- Investigating the mechanism of catalysis requires complementary spectroscopic techniques. Vibrational spectroscopies are often a method of choice to probe metal-adsorbate interactions. X-ray spectroscopy (XAS) is used to study bulk structure of the catalyst. While diffuse reflectance Fourier transform infrared spectroscopy (DRIFTS) is commonly used on a bench top, XAS requires synchrotron light. To merge the techniques for in-situ catalysis studies, an XAS /DRIFTS instrument was developed at NSLS beamline X18A.
- A rapid-scan FTIR spectrometer is outfitted with a range of detectors and beamsplitters for both mid- and far-infrared measurements, and is further equipped with a modified praying mantis DRIFTS attachment and a custom-made in situ reaction cell. The reaction cell can be heated to 800 ^{o}C, allows passage of gases through the catalyst, and is equipped with glassy carbon windows for x-ray light passage. Thus, both XAS and DRIFTS techniques can be done simultaneously with controlled conditions. Along with fast-moving monochromator for quick-EXAFS and mass-spectrometric residual gas analysis, this new tool is a powerful method for testing catalytic reactions in real time.



Experimental setup (left) and the reaction cell (insert in top right).
CO adsorption during the reduction of 5% Pt/γ-Al_2O_3 at elevated temperatures: as the Pt is reduced, which is monitored by XANES at the Pt L_3 edge (middle right), CO is observed to be adsorbed on Pt/γ-Al_2O_3 by DRIFTS (bottom right)

Accepted by *Nuclear Instruments and Methods in Physics Research, A*

Work performed on NSLS beamline X18A